MTL TR 91-19

2

ULTRASONIC EVALUATION OF SILICON NITRIDE GREEN BODIES

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May 1991

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 91-19	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* ULTRASONIC EVALUATION OF SILICON NITRIDE GREEN BODIES		5. TYPE OF REPORT & PERIOD COVERED Final Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR*(s)* Janet Quinn and Lisa Tardiff		8. CONTRACT OR GRANT NUMBER*(s)*
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-EMC		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE May 1991 13. NUMBER OF PAGES 12
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*		15. SECURITY CLASS. *(of this report)* Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT *(of this Report)* Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)* Ultrasonic Silicon nitride Green bodies		
20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)* (SEE REVERSE SIDE)		

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ABSTRACT

Advances in ultrasonic materials evaluation technology have greatly expanded the potential usefulness of this nondestructive technique to the ceramics industry. The development of dry coupling transducers has been especially beneficial to the early stages of ceramic processing with increasing application to green body evaluation.

In this study, ultrasonic techniques are utilized to determine property relationships in silicon nitride green bodies. A correlation of ultrasonic velocities with green body densities was found to be affected by a commercial plasticizer utilized in processing the slurries. This correlation was further investigated with photomicrographs, porosimetry, and a BET analysis.

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INTRODUCTION

The unique advantages of nondestructive ultrasonic evaluation have long been recognized and appreciated in the field of ceramic engineering. Nondestructive testing techniques have been utilized in evaluating sintered ceramics,[1-3] and more recently, to monitor slip casting rates.[4] The application of these techniques to ceramic green body evaluation, however, has been slowly pursued due to the fragileness of the prefired state. Such ceramics can quickly disintegrate under the minimal pressure employed with liquid couplants. Attenuation of the ultrasonic wave is also a problem with porous materials.

The original work in this area by researchers such as Jones, Blessing, and Robbins,[5] as well as Bhardwaj and Trippett,[6] and the current availability of dry transducers has greatly expanded the potential of ultrasonics in ceramic green body evaluation. Agglomeration content[5] and the influences of a binder in alumina green bodies[7] have been characterized by dry coupling techniques. These techniques have also been utilized to examine the property relationships of alumina and steatite green bodies.[6]

In this study, properties of slip cast silicon nitride green bodies are investigated. The effect of some common additives is examined and the role of nondestructive ultrasonic techniques in this evaluation is discussed. This work is part of a larger study investigating the slip casting of silicon nitride and the controlling parameters.

EXPERIMENTAL PROCEDURE

Thirteen 5.5 cm square plates were slip cast of silicon nitride with 6% yttria and 2% alumina by weight; a common sintering composition. Eleven plates were cast of UBE-SN-E-10* silicon nitride powder, the other two of TOSOH-TS-10.† These two powders have a similar chemical analysis, specific surface area, particle size distribution, and are synthesized by similar processing routes.

The powders were mixed with distilled water to form slurries with solids ratios of 54% to 62%. Ammonium hydroxide was used to control viscosities by varying the pH.

Four of the UBE plates contained Darvan C,†† a commercially available dispersant. Optimal slips contained 0.2% to 0.4% of the Darvan C solution. Four other UBE plates contained 0.6% Polyglycol E400,** a plasticizer.

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†TOSOH Corp., Nanyo Plant 4560, Sinnanyo-shi, Yamaguchi-ken 746, Japan

††R. T. Vanderbuilt, Co., Inc., 30 Winfield St., Norwalk, CT.

**Dow Chemical Co., Midland, MI.

1. NAGARAJAN, A. *Ultrasonic Study of Elasticity - Porosity Relationships in Polycrystalline Alumina.* J. App. Phys., v. 42, 1971, p. 3693.
2. ARONS, R. M., and KUPPERMAN, D. S. *Use of Sound Velocity Measurements to Evaluate the Effect of Hot Isostatic Pressing on the Porosity of Ceramic Solids.* Mater. Eval., v. 40, 1982, p. 1076.
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After drying the bodies for several days under ambient conditions, the plate faces were sanded flat to a final thickness between 0.8 cm and 1 cm. The green bodies were then heated in air to 700°C for one hour to drive off moisture and burn out the additives.

To examine the acoustic properties of the plates, an ultrasonic pulse echo contact system consisting of a 275 MHz bandwidth delaying time base oscilloscope and a high energy broadband pulser/receiver were used with selected low frequency transducers directly coupled to the plates.

Normally, with fully dense plates, a gel is used to couple the transducer to the plate surface in order to eliminate any air gaps. This provides better transmission of sound into the highly attenuating material. The dry coupling transducers utilized in this study have a thin, pliable surface which closely matches the impedance of the test material and provides the necessary transmission of sound without the liquid couplant a conventional transducer requires.

Five acoustic measurements for each plate were taken, one measurement near each of the four corners and one in the middle of the plate. These measurements consisted of longitudinal and shear times of flight in microseconds. A micrometer was used to measure the plate thickness at each of the five chosen points. The average acoustic measurements of each plate were later combined with plate density measurements to determine Young's modulus, E, and the shear modulus, G.

A mercury porosimeter was utilized for bulk density measurements and porosimetry curves were obtained for all samples. The low green densities limited specimen sizes to approximately one gram as the porosimeter sample tubes could only accommodate a volume of 0.6 cm^3 of intruded mercury.

It should be noted that the mercury porosimeter data for this study are not indicative of actual pore sizes since the Washburn equations assumptions[8] are invalid for such porous green bodies. It is believed the intrusion curves reflect the size distribution of the channels between voids.[9]

Parts of four specimens were shaped into small cylinders, about 0.9 cm diameter by 1.5 cm long, to fit into tubes for a BET analysis, performed by MRA Laboratories in North Adams, MA. Photomicrographs of the four specimens were also obtained.

RESULTS

Figure 1 indicates the modulus-density relationships. The data marked UBE and TOSOH are comprised of those starting powders and the data marked Darvan C and PEG contain the Darvan C and Polyglycol E400 additive, respectively. All slurries contain the yttria and alumina sintering aids.

The Figure 1 data follow the same straight lines with the exception of the specimens containing Polyglycol E400 which follow lower, parallel lines. The higher two lines with steep slopes are associated with Young's Modulus, E, on the ordinate, while the lower lines represent the shear modulus, G.

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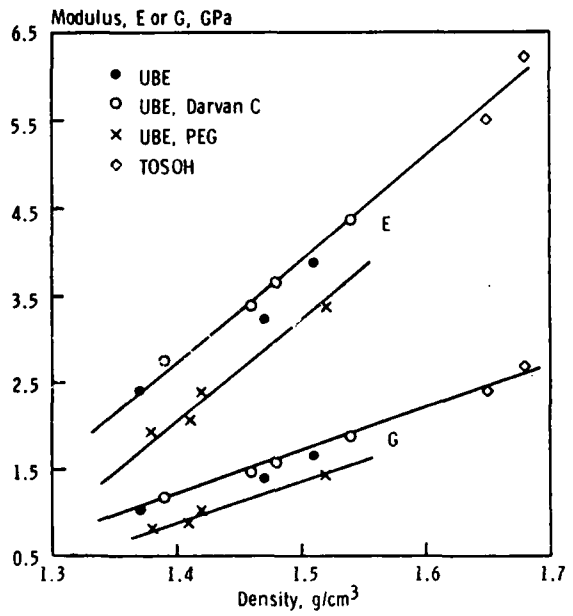

Figure 1. Modulus versus Density. The higher two lines are graphed with Young's modulus, E, on the ordinate, while the lower two lines are graphed with the shear modulus, G. All data follow common lines except the slurries which contained the polyglycol additive (UBE, PEG).

The formulae utilized in this study for determining the moduli from ultrasonic wave velocities are:[10]

$$E=\rho V_\ell^2 (1+\upsilon)(1-2\upsilon)/(1-\upsilon) \quad (1)$$

$$G=\rho V_t^2 \quad (2)$$

where

ρ = material density
V_ℓ = longitudinal wave velocity
V_t = transverse or shear wave velocity
υ = Poisson's ratio = (E/2G)-1.

Equation 1 and Equation 2 have often been utilized in characterizing fully dense materials. M. C. Bhardwaj[11] has demonstrated very good agreement between acoustically and mechanically (four-point bending) derived moduli in porous, and even ultra-porous, ceramics. The test pieces in the Reference 11 study, however, were sintered and more rigid than typically fragile ceramic green bodies. The exact relationship between acoustically and mechanically derived moduli in ceramic green bodies has not yet been thoroughly investigated.

It is interesting to note the acoustic moduli in these modulus versus density graphs are initially determined as functions of density. Thus, we have the same variable (density) represented on both the x- and y-axis. Eliminating this variable from the y-axis results in a potentially useful relationship for determining green body densities when a few constants have been previously obtained.

10. MCGONNAGLE, W. J. *Nondestructive Testing.* Gordon and Breach, Science Publishers, Inc., New York, N.Y., 1961, p. 211.
11. BHARDWAJ, M. C. *Simple Ultrasonic NDC for Advanced Ceramics Development and Manufacture.* Proceedings of International Process Modeling and Mechanical Behavior. Anaheim, CA, February 19-22, 1990.

From Figure 1:

$$G = m\rho + b$$

$$G = \rho V_t^2 \tag{2}$$

where m and b are the slope and y-intercept. Therefore,

$$\rho V_t^2 = m\rho + b$$

$$V_t^2 = b/\rho + m \tag{3}$$

Equation 3 indicates linear relationships between the shear velocities squared and reciprocal densities. These relationships are graphed in Figure 2 utilizing the Figure 1 data. The advantage in expressing the data in this fashion is that densities for these green bodies can now be predicted from shear velocities.

It is apparent from Figure 1 and Figure 2 that the specimens which were cast with the polyglycol additive resulted in green bodies slightly different from the others. To explore these differences two pairs of specimens, with and without the additive, were examined in greater detail. The first pair have similar green densities of 1.37 g/cm^3 and 1.38 g/cm^3. They comprise the photomicrographs in Figure 3a and Figure 3b. The specimen of Figure 3b contained the polyglycol, while the Figure 3a specimen did not. The second pair have green densities of 1.51 g/cm^3 and 1.52 g/cm^3 and comprise Figure 4a and Figure 4b, respectively. Figure 4b is the specimen processed with the additive. Close scrutiny of the photomicrographs reveal the specimens which contained the additive to be composed of

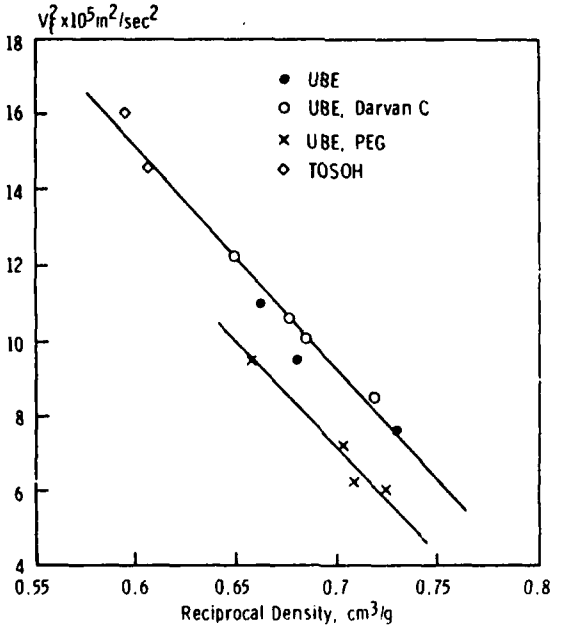

Figure 2. Shear velocity squared, V_t^2, versus reciprocal density. The slurries which were processed with the polyglycol additive fall on the lower line.

photomicrographs reveal the specimens which contained the additive to be composed of slightly more discrete, less agglomerated particles. This very slight difference is difficult to determine in the Figure 3 specimens, but is noticeable in Figure 4. Since more discrete particles within a green body should result in a higher internal surface area, a BET analysis was obtained for the four specimens of Figure 3 and Figure 4.

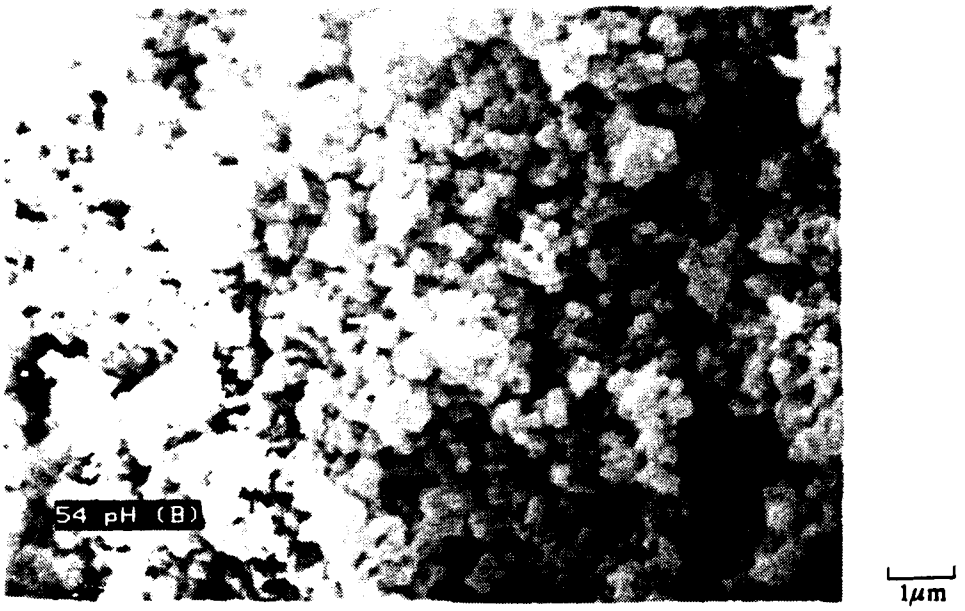

Figure 3a. Specimen without additive. Density = 1.37 g/cm^3.

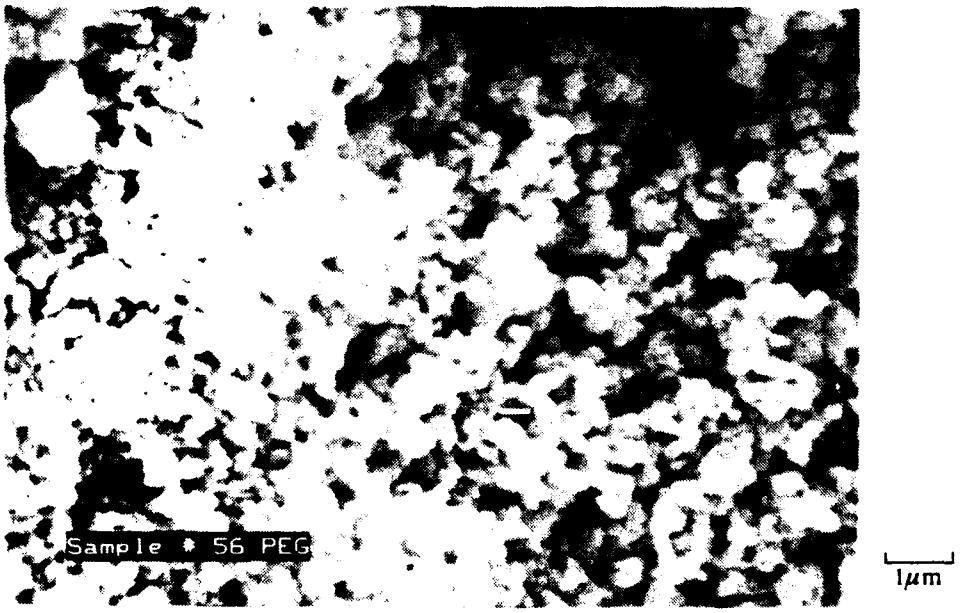

Figure 3b. Specimen with polyglycol additive. Density = 1.38 g/cm^3.

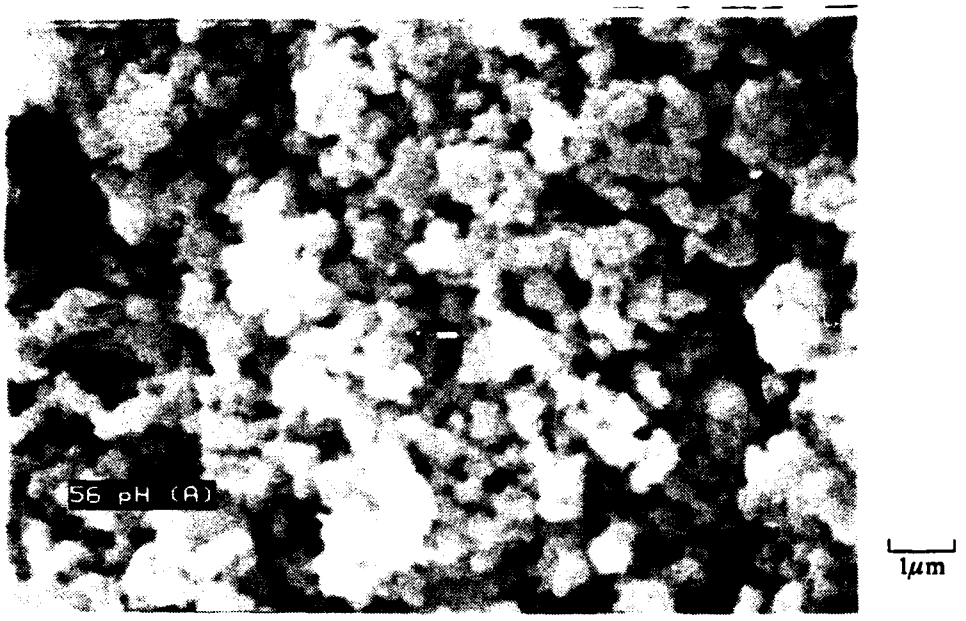

Figure 4a. Specimen without additive. Density 1.51 g/cm^3.

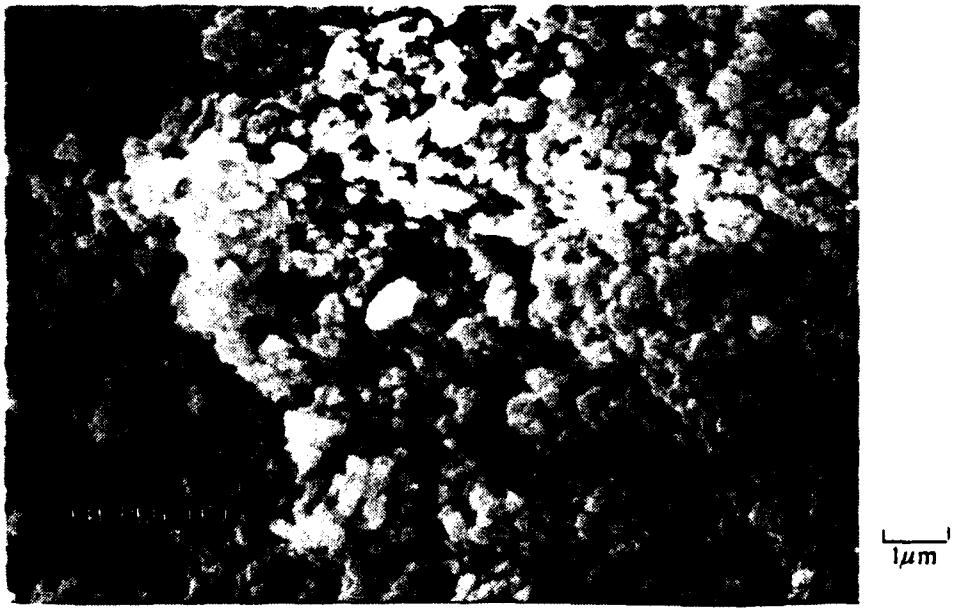

Figure 4b. Specimen with polyglycoladditive. Density = 1.52 g/cm^3.

The results of the BET analysis comprise Table 1. The error in internal surface area measurement is less than 5%, probably about 2% or 3%. The surface area of the initial starting powder is 11 m^2/g, by BET analysis provided by UBE Industries. The lower internal surface areas of the green bodies indicate the amount of particle joining which has occurred.

From Table 1, the surface areas of the specimens which contained the additive are slightly higher than the corresponding specimens which did not. This difference is more apparent in the Figure 4 specimens, and tends to confirm the observation of slightly more discrete particles within the samples which contained the polyglycol.

Table 1. BET INTERNAL SURFACE AREAS

Specimen Identification	Density (g/cm^3)	Additive Polyglycol E400	Surface Area (m^2/g)
Figure 3a	1.37	none	9.61
Figure 3b	1.38	0.6%	9.67
Figure 4a	1.51	none	9.47
Figure 4b	1.52	0.6%	9.75

Porosimeter analysis revealed no significant differences among all the specimens, the magnitude and shape of the mercury intrusion curves being consistent with specimen size and density. As the specimen densities increased, the porosimeter curves began rising at higher pressures. This corresponds to the increased difficulty of the mercury in penetrating smaller spaces between particles. The curves also flattened out more gradually as the specimen densities increased, indicating a broader size distribution of the spaces between particles. These observations are illustrated by the two intrusion curves of Figures 5a and Figure 5b. Higher pressures were necessary for the mercury to penetrate the higher density specimen of Figure 5b.

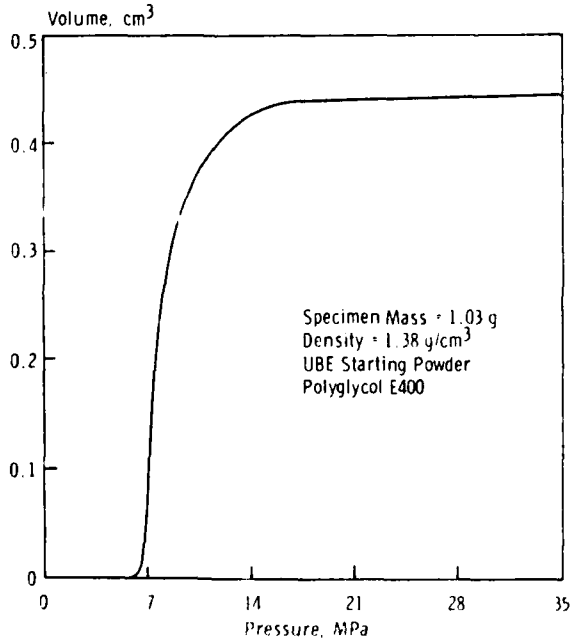

Figure 5a. Intruded Hg volume versus pressure for a low density sample.The low pressure at which intrusion begins (6.9 MPa) followed by rapid complete intrusion is typical of the low density green bodies of this study.

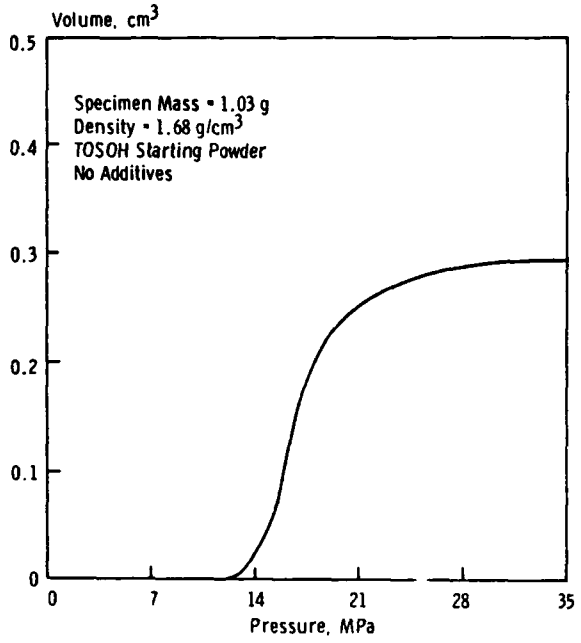

Figure 5b. Intruded Hg volume versus pressure for a higher density sample. Mercury intrusion begins at a higher pressure (13.8 MPa) and levels off more slowly, indicating the narrower and more distributed particle spaces typical of the higher density green bodies of this study.

The pressures at which the most mercury intrusion takes place are indicative of the median space sizes between particles. These "median" pressures were graphed against specimen densities in Figure 6. The resulting curve is fairly consistent for all the specimens, with and without additives. This curve becomes very steep as the specimen densities increase; in the limit, channel sizes would become very small and close up entirely.

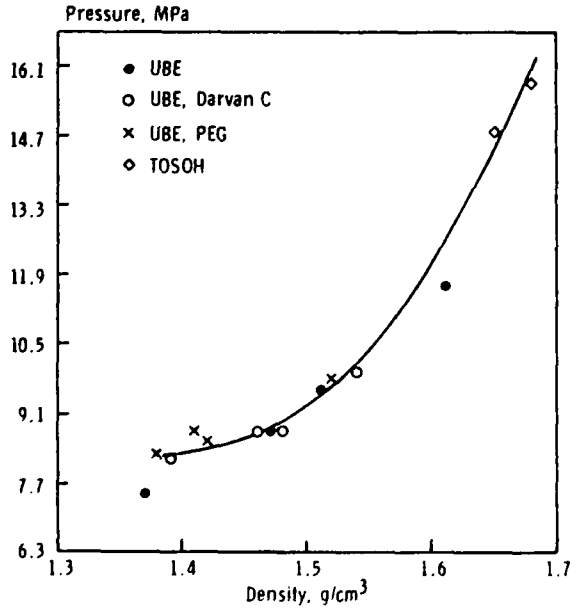

Figure 6. Intrusion pressure versus density. The mercury intrusion pressures rise more quickly with density as the channels between particles become narrower.

DISCUSSION

The linear relationship of the densities and ultrasonically determined moduli in silicon nitride green bodies is not surprising; such linear relationships in other ceramic green bodies have been documented.[6] Ultrasonic techniques were utilized in this study to observe any change, or lack of it, when the green bodies were processed differently.

There appear to be no discernable differences among the specimens processed with and without the Darvan C additive. The primary effect of Darvan C was to lower the pH values at which the slips could achieve low viscosities.

There is a noticeable change in the ultrasonic behavior of the specimens processed with the polyglycol additive. Such specimens have lower ultrasonic moduli than specimens processed without this additive. Since the material composition and densities are the same, there are possibly structural differences in these green bodies.

Comparisons of specimens of similar densities by means of photomicrographs, porosimetry, and BET analysis are not as conclusive as the ultrasound evaluation, although subtle changes in the photomicrographs are weakly confirmed by Table 1. It is possible the polyglycol additive coated the particles, causing them to remain separated after the additive was burned out. The ultrasound analysis is apparently more sensitive to changes wrought by such additives.

It was noted during the processing of the polyglycol samples that it was necessary to significantly increase the slurry solids concentrations in order to obtain green bodies of similar densities as those processed without polyglycol. Thus, a slurry containing 60% solids with Polyglycol E400 resulted in a green body with a similar density as one containing only 56% solids without this additive. As polyglycol can be used to increase green body strength and machinability before burnout, the amount and desirable effects must be weighed against a possible lower green body density after burnout. There also remain questions as to how the differences indicated by the ultrasound analysis might affect sinterability.

The total volumes of intruded mercury were found to equal the air volumes in the specimens calculated through density measurements to within 1%. It can therefore be concluded that all the pores in these green bodies are surface connected. BET analysis on such specimens should reflect true internal areas.

CONCLUSION

Silicon nitride green body densities are strongly and clearly related to ultrasonic velocities. This relationship can be expressed as a linear function with the linear constants being a function of processing variables. Ultrasonic testing, therefore, can be used as an effective means of determining green body densities in previously tested processing compositions.

In this study, green bodies processed with a polyglycol additive showed differences not readily apparent with other testing techniques, while those processed with a commercial dispersant exhibited no such differences. These differences are probably structural and could affect subsequent sintering.

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Technical Report MTL TR 91-19, May 1991, 12 pp-illus-tables

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Advances in ultrasonic materials evaluation technology have greatly expanded the potential usefulness of this nondestructive technique to the ceramics industry. The development of *dry coupling transducers has been especially beneficial to the early stages of ceramic pro*cessing with increasing application to green body evaluation. In this study, ultrasonic techniques are utilized to determine property relationships in silicon nitride green bodies. A correlation of ultrasonic velocities with green body densities was found to be affected by a commercial plasticizer utilized in processing the slurries. This correlation was further investigated with photomicrographs, porosimetry, and a BET analysis.

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ULTRASONIC EVALUATION OF SILICON NITRIDE GREEN BODIES
Janet Quinn and Lisa Tardiff

Technical Report MTL TR 91-19, May 1991, 12 pp-illus-tables

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